# LABORATORY ACTIVITIES

## INTRODUCTION

On June 19, 20 and 21, 2017 the U.S. Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) laboratory received 69 wastewater samples from nine stations and six trip blanks all collected in June 2017 as part of a focused clean air act compliance investigation of 3V Inc, located in Georgetown, South Carolina. Laboratory sample receipt and inspection information are summarized in **Table 1**. The samples were maintained under custody in accordance with the NEIC operating procedure *Evidence Management*, NEICPROC/00-059R4.

**Table 1. LABORATORY SAMPLE RECEIPT AND INSPECTION**
**3V**
**Georgetown, South Carolina**

| Event | Date | Comments |
|---|---|---|
| Samples received | June 19, 2017 | Delivered to NEIC by FedEx (tracking No.7389 6763 2065) and received by chemist Robyn Hallowell (R. Hallowell). |
| Samples unpacked and inspected | June 19, 2017 | Shipping container was a red cooler secured with packing tape. A padlock/hasp assembly on the front was dislodged during shipping. The packing tape was still secure.<br><br>R. Hallowell unpacked and inspected the contents of the red cooler, which included four aluminum evidence cans. In the cans, sealed plastic baggies with water mix and samples. Cooler contained 40-milliliter (mL) volatile vial samples. The chain of custody paperwork was taped inside the cooler. All sample information was verified against the chain of custody record (No. 14266), and the samples were placed into R. Hallowell's cart, which was then locked, and stored in walk-in cooler B in room 1C-210. Upon the full completion of inspection, the chain of custody was signed. Samples were given to PAC Angie Hunter (A. Hunter) June 20, 2017. |
| Samples received | June 20, 2017 | Delivered to NEIC by UPS (tracking No. 1Z A42 01T 84 9206 9952) and received by chemist A. Hunter. |
| Samples unpacked and inspected | June 20, 2017 | Shipping container was a blue cooler secured with a padlock/hasp assembly on the front.<br><br>A. Hunter unpacked and inspected the contents of the blue cooler. Cooler contained 40-mL volatile vial samples, and field sample supplies. The chain of custody paperwork was taped inside the cooler. All the samples were sealed in custody bags. All security seals were intact. All sample information was verified against the chain of custody record (No. 14265), and the samples were placed into A. Hunter's cart, which was then locked, and stored in walk-in cooler B in room 1C-210. |
| Samples received | June 21, 2017 | Delivered to NEIC by UPS (tracking No. 1Z A42 01T 84 9445 5110) and received by chemist A. Hunter. |

**Table 1. LABORATORY SAMPLE RECEIPT AND INSPECTION**
**3V**
**Georgetown, South Carolina**

| Event | Date | Comments |
|---|---|---|
| Samples unpacked and inspected | June 21, 2017 | Shipping container was a blue cooler secured with a padlock/hasp assembly on the front.<br><br>A. Hunter unpacked and inspected the contents of the blue cooler. Cooler contained 40-mL volatile vial samples, and field sample supplies. The chain of custody paperwork was taped inside the cooler. All the samples were sealed in custody bags. All security seals were intact. All sample information was verified against the chain of custody record (No. 14267), and the samples were placed into A. Hunter's cart, which was then locked, and stored in walk-in cooler B in room 1C-210. |

## ANALYTICAL PROCEDURES AND ANALYSTS

**Table 2** lists the analytical procedures and analysts. All analyses were conducted by NEIC personnel in accordance with the NEIC quality system from June through July 2017. Data quality summaries for all laboratory measurements are maintained in the project file. All laboratory procedures cited in this report are within the scope of NEIC's ISO/IEC 17025 accreditation issued by the ANSI-ASQ National Accreditation Board (certificate No. AT-1646).

**Table 2. ANALYTICAL PROCEDURES AND ANALYSTS**
**3V**
**Georgetown, South Carolina**

| Analytical Method/Procedures | Analyst |
|---|---|
| • EPA SW-846 Method 5030B, *Purge-and-Trap for Aqueous Samples*<br>• EPA SW-846 Method 8260B, *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*<br>• *Volatile Organic Analysis by Gas Chromatography/Mass Spectrometry*, NEICPROC/00-002R5 | Angie Hunter |
| • EPA SW-846 5021A*, Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis*<br>• *Volatile Organic Analysis by Gas Chromatography/Mass Spectrometry*, NEICPROC/00-002R5 | Robyn Hallowell |

## ANALYTICAL RESULTS

The wastewater samples, were analyzed by A. Hunter and R. Hallowell for the presence of ethylbenzene, methanol, methylene chloride, and xylenes using gas chromatography/mass spectrometry (GC/MS). **Table 3** summarizes the analyte concentrations determined in the samples and their associated limit of quantitation (LOQ). The LOQ values were based on the sample preparation, along with the most dilute standard solution used in generating the calibration curve for analyte quantitation.

The estimated measurement uncertainty values for analytes summarized in **Table 3** were based on standard data for ethylbenzene (±16%), methylene chloride (±10%), and xylenes (±17%). For methanol (±10%), the uncertainty was based on the highest RSD of triplicates. However, at the lowest concentration, the uncertainty becomes larger.

**Table 3. WASTEWATER SAMPLE RESULTS**
**3V, Inc.**
**Georgetown, South Carolina**

| Station # | NEIC Tag# | Analyte | Analyte Concentration ($mg/L^3$) | Limit of Quantitation (LOQ) (mg/L) |
|---|---|---|---|---|
| 1[1] | NE39152 | Methylene Chloride | 1,090,000[4] | 400,000 |
| | NE39201 | Methylene Chloride | 1,150,000[4] | 400,000 |
| 2[1] | NE39157[2] | Ethylbenzene | 34.1 | 5 |
| | | m-&/or p-Xylene | 142 | 5 |
| | | Methylene Chloride | 64.8 | 5 |
| | | o-Xylene | 43.4 | 5 |
| | NE39159[2] | Methanol | < LOQ | 14 |
| 2 | NE39214 | Ethylbenzene | 34.6 | 5 |
| | | m-&/or p-Xylene | 145 | 5 |
| | | Methylene Chloride | 69.2 | 5 |
| | | o-Xylene | 43.6 | 5 |
| | NE39216[2] | Methanol | < LOQ | 14 |
| 3[1] | NE39161 | Ethylbenzene | 2.84 | 0.5 |
| | | m-&/or p-Xylene | 10.8 | 0.5 |
| | | Methylene Chloride | 23.8 | 0.5 |
| | | o-Xylene | 3.77 | 0.5 |
| | NE39163[2] | Methanol | 62 | 14 |
| | NE39205 | Ethylbenzene | 9.73 | 1 |
| | | m-&/or p-Xylene | 41.8 | 1 |
| | | Methylene Chloride | < LOQ | 1 |
| | | o-Xylene | 13.7 | 1 |
| | NE39207[2] | Methanol | < LOQ | 14 |
| 4[1] | NE39165 | Ethylbenzene | 12.2 | 1 |
| | | m-&/or p-Xylene | 53.2 | 1 |
| | | Methylene Chloride | 20.4 | 1 |
| | | o-Xylene | 18.3 | 1 |
| | NE39167[2] | Methanol | 92 | 14 |
| 5[1] | NE39169 | Ethylbenzene | 3.11 | 0.5 |
| | | m-&/or p-Xylene | 13.0 | 0.5 |
| | | Methylene Chloride | 21.2 | 0.5 |
| | | o-Xylene | 4.1 | 0.5 |
| | NE39171[2] | Methanol | 350 | 14 |
| | NE39189[2] | Ethylbenzene | 2.85 | 0.5 |
| | | m-&/or p-Xylene | 10.6 | 0.5 |
| | | Methylene Chloride | 18.8 | 0.5 |
| | | o-Xylene | 4.11 | 0.5 |
| | NE39191[2] | Methanol | 220 | 14 |
| 6[1] | NE39175 | Ethylbenzene | < LOQ | 0.05 |
| | | m-&/or p-Xylene | 0.167 | 0.05 |
| | | Methylene Chloride | < LOQ | 0.05 |
| | | o-Xylene | < LOQ | 0.05 |
| | NE39177[2] | Methanol | 1,500 | 140 |
| 6 | NE39218 | Ethylbenzene | < LOQ | 0.05 |
| | | m-&/or p-Xylene | 0.114 | 0.05 |

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**3V, Inc.**
**Georgetown, South Carolina**

| Station # | NEIC Tag# | Analyte | Analyte Concentration ($mg/L^3$) | Limit of Quantitation (LOQ) (mg/L) |
|---|---|---|---|---|
| | | Methylene Chloride | < LOQ | 0.05 |
| | | o-Xylene | < LOQ | 0.05 |
| | NE39220[2] | Methanol | 95,000 | 14,000 |
| 7[1] | NE39179 | Ethylbenzene | < LOQ | 50 |
| | | m-&/or p-Xylene | 98.8 | 50 |
| | | Methylene Chloride | 391 | 50 |
| | | o-Xylene | < LOQ | 50 |
| | NE39180 | Ethylbenzene | < LOQ | 50 |
| | | m-&/or p-Xylene | 100 | 50 |
| | | Methylene Chloride | 343 | 50 |
| | | o-Xylene | < LOQ | 50 |
| | NE39181 | Ethylbenzene | < LOQ | 50 |
| | | m-&/or p-Xylene | 92.9 | 50 |
| | | Methylene Chloride | 296 | 50 |
| | | o-Xylene | < LOQ | 50 |
| | NE39184 | Methanol | 650 | 28 |
| | NE39185 | Methanol | 640 | 28 |
| | NE39186 | Methanol | 660 | 28 |
| 7 | NE39210 | Ethylbenzene | 84.9 | 50 |
| | | m-&/or p-Xylene | 376 | 50 |
| | | Methylene Chloride | 1220 | 50 |
| | | o-Xylene | 93.3 | 50 |
| | NE39212[2] | Methanol | 110 | 14 |
| 8[1] | NE39193 | Ethylbenzene | < LOQ | 5 |
| | | m-&/or p-Xylene | < LOQ | 5 |
| | | Methylene Chloride | < LOQ | 5 |
| | | o-Xylene | < LOQ | 5 |
| | NE39195[2] | Methanol | 460 | 14 |
| 8 | NE39222[2] | Ethylbenzene | < LOQ | 5 |
| | | m-&/or p-Xylene | < LOQ | 5 |
| | | Methylene Chloride | 8.17 | 5 |
| | | o-Xylene | < LOQ | 5 |
| | NE39224[2] | Methanol | 99 | 14 |
| 9[1] | NE39197 | Ethylbenzene | 234 | 50 |
| | | m-&/or p-Xylene | 1070 | 50 |
| | | Methylene Chloride | 70.1 | 50 |
| | | o-Xylene | 343 | 50 |
| | NE39199[2] | Methanol | 33 | 14 |
| Trip Blank[1] | NE39155 | Ethylbenzene | < LOQ | 0.001 |
| | | m-&/or p-Xylene | < LOQ | 0.001 |
| | | Methylene Chloride | 0.006 | 0.001 |
| | | o-Xylene | < LOQ | 0.001 |
| | | Methanol | < LOQ | 14 |
| Trip Blank[1] | NE39172 | Ethylbenzene | < LOQ | 0.001 |
| | | m-&/or p-Xylene | < LOQ | 0.001 |
| | | Methylene Chloride | < LOQ | 0.001 |
| | | o-Xylene | < LOQ | 0.001 |
| Trip Blank[1] | NE39173 | Ethylbenzene | < LOQ | 0.001 |
| | | m-&/or p-Xylene | < LOQ | 0.001 |
| | | Methylene Chloride | < LOQ | 0.001 |

**Table 3. WASTEWATER SAMPLE RESULTS**
**3V, Inc.**
**Georgetown, South Carolina**

| Station # | NEIC Tag# | Analyte | Analyte Concentration ($mg/L^3$) | Limit of Quantitation (LOQ) (mg/L) |
|---|---|---|---|---|
| | | o-Xylene | < LOQ | 0.001 |
| | | Methanol | < LOQ | 14 |
| Trip Blank[1] | NE39187 | Ethylbenzene | < LOQ | 0.001 |
| | | m-&/or p-Xylene | < LOQ | 0.001 |
| | | Methylene Chloride | < LOQ | 0.001 |
| | | o-Xylene | < LOQ | 0.001 |
| | | Methanol | < LOQ | 14 |
| Trip Blank[1] | NE39208 | Ethylbenzene | < LOQ | 0.001 |
| | | m-&/or p-Xylene | < LOQ | 0.001 |
| | | Methylene Chloride | 0.001 | 0.001 |
| | | o-Xylene | < LOQ | 0.001 |
| | | Methanol | < LOQ | 14 |
| Trip Blank | NE39225 | Ethylbenzene | < LOQ | 0.001 |
| | | m-&/or p-Xylene | < LOQ | 0.001 |
| | | Methylene Chloride | < LOQ | 0.001 |
| | | o-Xylene | < LOQ | 0.001 |
| | | Methanol | < LOQ | 14 |

[1] 14 day hold time missed or samples did not come in on ice. Concentrations should be considered a minimum value.
[2] Results are from triplicate tests; n = 3.
[3] mg/L = milligrams per liter.

[4] Determined concentrations above 100% are due to the dilution ratios of the samples.